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# *The Electrolysis of Metallic Formates.*

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*(Read before the American Philosophical Society, November 6, 1891.)*

The facility with which many metallic formates could be reduced to the metallic state by heat, or in the case of silver and mercury, even by the action of light, having led to the hope that they might be employed with particular advantage in electrolysis, the following series of experiments were made upon solutions of copper, zinc and cadmium formates, in order to ascertain the effect of dilution, temperature and pole separation, as well as the conditions necessary in order to effect their quantitative estimation and separation. The current was generated by a battery of ten cells, of the "crowfoot" type, each cell being 3.1 dm. in height, by 1.9 dm. in diameter, and having a capacity of 2 liters; the dimensions of the zincs were 1.5 cm. by 1.5 cm., and of the radiating copper plates constituting the positive pole 1.5 cm. by 1.5 cm. By means of this battery a comparatively uniform current of 2.8 c.c. electrolytic gas per minute was generated after the cells had been in use for some time.

The strength of the current was measured by means of an ordinary voltameter, and was ascertained before and after the completion of the experiment. For the deposition of small quantities of metal, thick platinum-foil electrodes were used, 3.8 cm. wide, and immersed to the depth of 3.8 cm. in the solution. For quantities above .05 gram, they were unsatisfactory, the metal showing a great tendency to separate in a spongy condition at the edge. In the earlier determinations a platinum dish was used, weighing about 67 grams, and having a capacity of 150 c.c.; in the later ones a dish weighing 117 grams, and with a capacity of 275 c.c., was employed. The results obtained with the larger dish were necessarily somewhat less exact than with the one of smaller size. The positive pole consisted of a thick platinum wire, the lower portion of which was wound into a horizontal spiral. In some of the separations it was found expedient to substitute for the spiral a small platinum crucible 2.5 cm. in height and 2.8 cm. in diameter, closed by a cork, through which passed a copper wire in contact with the bottom of the crucible. In order to regulate the distance between the poles, a filter stand was used, having inserted on its movable arm an ordinary binding screw, to which the positive pole was attached.

The following formates were prepared:

## COPPER FORMATE.

This salt was made by precipitating cupric oxide from a hot solution of copper sulphate, by means of caustic potash; the precipitate was washed by decantation until free from traces of potash; it was then dissolved in formic acid having the sp. gr. 1.015, obtained in the usual way from oxalic acid and glycerine, through which a current of steam was allowed to pass in order to prevent too great a rise of temperature, with the consequent production of decomposition products; the salt was allowed to crystallize out by spontaneous evaporation in a current of warm air, and recrystallized. An abundant crop of large, blue, monoclinic crystals was obtained, having the composition $Cu\,(C\,HO_2)^2 + 4\,H_2O$, efflorescing in dry air, soluble in eight parts of water and changed by boiling to the sparingly soluble basic salt $Cu\,(C\,HO_2)^2.\ 2\,Cu\,(HO)^2$.

### ZINC FORMATE.

A solution of ordinary crystallized zinc sulphate was treated with an excess of sodium carbonate, heated almost to boiling, freed by decantation from soluble impurity and dissolved in hot formic acid. The solution was evaporated down and allowed to stand, after filtering off a slight precipitate that formed on boiling, and which gave the iron reaction with potassium sulphocyanate.

Monoclinic prisms having the formula $Zn\,(C\,HO_2)^2 + 2\,H_2O$ separated out, isomorphous with the cadmium salt, permanent in the air and soluble in twenty-four parts of water at ordinary temperature.

### CADMIUM FORMATE.

This salt was prepared by dissolving cadmium obtained by distillation in vacuuo, in nitric acid, neutralizing with a hot solution of potassium carbonate, washing by decantation until free from soluble carbonate and dissolving in formic acid. Large monoclinic crystals separated out, having the composition $Cd\,(C\,HO_2)^2 + 2\,H_2O$, permanent in the air, readily soluble in water.

(NOTE.—The formulas of copper and cadmium formates are given as follows: $Cu\,(C\,HO_2)^2$ and $Cd\,(C\,HO_2)^2$ in the last edition of Watts' *Dictionary of Chemistry*, differing from all other authorities. Experiments made to settle the question resulted in the formulas assigned, which is in accordance with the ones usually given.)

### LEAD FORMATE.

Solutions of lead acetate and sodium formate were mixed and allowed to stand. Large white anhydrous rhombic crystals gradually separated out in radiating needles, sparingly soluble in cold water, more readily in hot, but with partial decomposition into free acid, and a basic salt of variable composition.

### COBALTOUS FORMATE.

A hot solution of cobaltous sulphate was neutralized with caustic soda solution, washed by decantation until free from all but traces of the precipitant, dissolved in formic acid, filtered, and allowed to evaporate in a current of warm air. The salt separated in crusts, consisting of indistinct crystals, having the composition $Co\,(C\,HO_2)^2 + 2\,H_2O$, dissolving with difficulty to a reddish-colored solution.

## MANGANOUS FORMATE.

This salt was prepared from manganous carbonate precipitated from a hot solution of manganous sulphate, by means of sodium carbonate added to alkaline reaction and decanted as rapidly as possible until free from all except very slight traces of soluble salts. It was then dissolved in formic acid and allowed to crystallize very slowly. The crystals thus obtained were allowed to recrystallize. The crystals are small, pale reddish monoclinic prisms, soluble in fifteen parts of water, and contain two molecules of water of crystallization.

## NICKEL FORMATE.

A solution of nickel chloride was treated with a slight excess of sodic hydrate, washed several times by decantation with hot water, dissolved in acid and evaporated down. A greenish crust formed, made up of very small, bright, green needles—$Ni\,(C\,HO_2)^2 + 2\,H_2O$.

## FERRIC FORMATE.

Ferric chloride was treated with excess of a solution of ammonia, washed with hot water, and allowed to digest in formic acid at a temperature which was not allowed to exceed 70°, until the hydrate of iron had completely dissolved, which required several hours. The deep-red solution was allowed to crystallize by spontaneous evaporation. Yellowish-red needles, crystallizing in radiating tufts, separated out, which formed a light, loose, coherent powder. When dried at a moderate temperature, it was readily soluble in cold water with an acid reaction. Aqueous solutions on warming became turbid from the partial decomposition of the salt into ferric hydrate and free acid. A similar decomposition takes place in solutions at ordinary temperatures after standing for some time. (The foregoing salt was made in preference to ferrous formate on account of its greater solubility.)

## MERCURIC FORMATE.

Mercuric oxide was dissolved in formic acid, but on warming the solution slightly it decomposed into the very sparingly soluble white mercurous formate, carbon dioxide and formic acid, according to the following equation :

$$2\,Hg\,(C\,HO_2)^2 = Hg_2\,(C\,HO_2)^2 + C\,HO_2H + CO_2 \text{ and}$$
$$Hg_2\,(C\,HO_2)^2 = 2\,Hg + C\,HO_2\,H + CO_2.$$

The precipitate was gray in color from the presence of free mercury. The tendency to decompose is such that in solution at ordinary temperatures these changes take place readily in the light and, with more slowness, even in the dark. The "ous" salt comes out in minute shining crystals, very insoluble in water, and on continuous warming becomes entirely converted into free mercury. The formates of silver, bismuth and tin were not prepared, as they were not considered available for various reasons.

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In order to ascertain the comparative accuracy of the results obtained by means of the ordinary gravimetric methods, as compared with those obtained by means of the current, a series of experiments was made with the metals chosen for particular study. The gravimetric method adopted was the same for all three, namely, estimation as oxide, conducted in the ordinary way.

### COPPER (DETERMINED AS CuO).

| Copper formate taken, in grams. | Copper by theory, in grams. | Copper in CuO formed. | Difference in percentage from theoretical. |
|---|---|---|---|
| (1) .8024 | .2252 | .2261 | +.39 per cent. |
| (2) .7924 | .2226 | .2232 | +.27 " |
| (3) .7063 | .1984 | .1982 | —.10 " |
| (4) .7063 | .1984 | .1979 | —.25 " |

The first two determinations were high, due perhaps to the efflorescence of the salt. All subsequent weighings were done in a covered watch-glass, and the results obtained corresponded closely with the theoretical.

In the following electrolytic depositions of copper, solutions of copper formate of known strength were used.

### COPPER (DETERMINED ELECTROLYTICALLY).

| Copper present in solution. | Copper found. | Free formic acid. | c.c. $H_2O$. | Time in hours. | Differ'e in percentage from theoretical. |
|---|---|---|---|---|---|
| (1) .1434 grams. | .1438 | 10 c.c. | 125 | 46 | +.27% |
| (2) .1074 " | 1075 | 15 c.c. | " | 16 | +.09 |
| (3) .0987 " | .0988 | 5 c.c. | " | 17 | +.10 |
| (4) .0987 " | .0987 | 10 c.c. | " | 16 | .00 |
| (5) .1074 " | .1077 | 10 c.c. | " | 16 | +.27 |
| (6) .1057 " | .1056 | 10 c.c. | " | 17 | —.09 |
| (7) .1057 " | .1052 | 15 c.c. | " | 17 | —.47 |
| (8) .1101 " | .1104 | 10 c.c. | " | 42 | +.27 |

In the above experiments a platinum dish was used at the negative pole, and the wire spiral as the anode. A current of 0.8—1.60 c.c. HO gas per minute was allowed to run over night. The poles were separated 2.5 cm. Before cutting off the current, the level of the liquid in the dish was raised by the addition of water, and the current allowed to act for an additional half hour. No further deposit of copper took place on the clean surface of the dish, indicating that the metal was completely precipitated. The current was then discontinued, the liquid quickly poured off, and the dish washed with hot water, being finally dried on a warm iron plate at a temperature not exceeding 100° C. The dish was then set aside for some time until it had acquired the temperature of the room and weighed. The solutions failed to give any indication, except the merest traces, of copper when tested with ferrocyanide of potassium. The time varied from sixteen to forty-six hours without materially affecting the result. No perceptible oxidation took place during drying, although the deposit was somewhat dark. It came out as a compact adherent coating, readily dissolving in nitric acid.

A comparison between the results obtained shows conclusively not only that the deposition of copper from its formate solution can be accomplished, but that it is fully as accurate as the ordinary gravimetric method.

### Zinc (Determined as ZnO).

| Zinc formate taken, in grams. | Zinc by theory, in grams. | Zinc in zinc oxide found. | Difference in percentage from theoretical. |
|---|---|---|---|
| (1) .5508 | .1875 | .1870 | —.26% |
| (2) " | " | .1876 | +.05 |
| (3) " | " | .1869 | —.31 |
| (4) " | " | .1872 | —.16 |

The four determinations made by the usual gravimetric method were estimated finally as zinc oxide which, for purposes of comparison, have been converted into metal.

### Zinc (Determined Electrolytically).

| Zinc present in grams. | Zinc found. | Free acid. | c.c.$H_2O$. | Time in hours. | Current in HO gas per minute. | Difference in percentage from theory. |
|---|---|---|---|---|---|---|
| (1) .0625 | .0614 | none | 100 | 16 | 1.8 c.c. | .... |
| (2) .0625 | .... | 10 c.c. | 100 | 16 | 1.8 c.c. | .... |
| (3) .1250 | .... | 15 c.c. | 100 | 16 | 2.0 c.c. | .... |
| (4) .0818 | .0476 | 5 c.c. | 100 | 17 | .8 c.c. | .... |
| (5) .0818 | .0816 | 15 c.c. | 100 | 17 | 1.7 c c. | —.24% |
| (6) .0818 | .0819 | 15 c.c. | 100 | 16 | 1.7 c.c. | +.12 |
| (7) .0513 | .0513 | 25 c.c. | 100 | 16 | 1.7 c.c. | .... |
| (8) .1026 | .1021 | 10 c.c. | 100 | 17 | 1.7 c.c. | —.48 |
| (9) .1026 | .1023 | 15 c.c. | 100 | 43 | 1.8 c.c. | —.29 |
| (10) .1006 | .1007 | 10 c.c. | 100 | 16 | 1.7 c.c. | +.09 |
| (11) .1006 | .1003 | 10 c.c. | 100 | 16 | 1.7 c.c. | —.29 |

The foregoing determinations of zinc formate were performed under a variety of conditions. (1), (2), (3), (4) were made with the dish as cathode, and the wire spiral as the positive pole; the results both with and without free acid were unsatisfactory, the deposit being very spongy and failing to come out completely, as proved by testing the solution with potassium ferrocyanide. The dish was then made the anode and the zinc was allowed to separate on the platinum crucible which was made the negative pole. The amount of free acid varied from 10 to 25 c.c. The deposit was gray and adherent on the sides and bottom, but rather spongy at the periphery. Around the top of the crucible the metal was black in color. The final determinations were accurate but required the greatest care to avoid detaching loose particles of metal.

The deposit was not regular, the bottom of the crucible being more or less free from zinc on account of the accumulation of gas. The time of deposition averaged sixteen hours. Such currents as sufficed to separate copper were unsatisfactory, even when the electrodes were brought in close contact. In the determinations that were satisfactory, the current strength varied from 1.6—1.8 HO gas per minute, and the poles were close together.

### CADMIUM (DETERMINED AS CdO).

| Cadmium formate taken. | Cadmium by theory. | Cadmium in CdO found. | Difference in percentage from theory. |
|---|---|---|---|
| (1) .4224 | .1988 | .1983 | —.25% |
| (2) .4224 | .1988 | .1982 | —.30 |

Both results are somewhat too low, possibly owing to reduction of the oxide to metal and consequent loss by volatilization.

### CADMIUM (DETERMINED ELECTROLYTICALLY).

| Cadmium present in grams. | Cadmium found in grams. | Free acid. | c.c. $H_2O$. | Time in hours. | Differ'ce in percent from theoretical. |
|---|---|---|---|---|---|
| (1) .0497 | .0498 | 15 c.c. | 100 | 16 | +.20% |
| (2) .0994 | .0996 | 10 c.c. | 100 | 18 | +.20 |
| (3) .0994 | .0991 | 10 c.c. | 100 | 16 | —.30 |
| (4) .1231 | .1228 | 10 c.c. | 100 | 44 | —.40 |
| (5) .1231 | .1229 | 10 c.c. | 100 | 17 | —.16 |
| (6) .0984 | .0984 | 10 c.c. | 100 | 16 | .... |
| (7) .0984 | .0985 | 10 c.c. | 100 | 16 | +.10 |
| (8) .1004 | .1005 | 10 c.c. | 100 | 16 | +.09 |
| (9) .1004 | .1002 | 10 c.c. | 100 | 17 | —.19 |

The dish was used as the negative electrode, the spiral as the positive, except (1), in which the cadmium was deposited on the crucible, the dish serving as the anode. The distance between the poles was 2.5 cm. The variations in the conditions of the experiments noted above caused no noticeable difference in the results. The deposit was not apparently oxidized by moderate warming. Current 1.25 to 1.5 c.c. HO gas per minute.

The solutions were tested for cadmium at the conclusion of each experiment, but none was found, proving that the metal was completely deposited. It formed a firm and adherent coating, white in color, with a bright metallic lustre.

## I. INFLUENCE OF DILUTION UPON THE PRECIPITATION OF COPPER.

| Copper present in grams. | Copper found. | Free acid. | c.c. $H_2O$. | c.c. HO gas per minute. | Time in hours. |
|---|---|---|---|---|---|
| (1) .0717 | .0199 | 3 drops | 100 | 1.75 | 1 |
| (2) .0358 | .0111 | $\frac{3}{2}$ " | " | " | " |
| (3) .0179 | .0057 | $\frac{3}{4}$ " | " | " | " |
| (4) .0089 | .0028 | $\frac{3}{8}$ " | " | " | " |
| (5) .0044 | .0014 | $\frac{3}{16}$ " | " | " | " |
| (6) .0022 | .0006 | $\frac{3}{32}$ " | " | " | " |
| (7) .0011 | .0002 | $\frac{3}{64}$ " | " | " | " |

The distance between the poles was 2.5 cm. The area of the electrodes was (3.8 cm. × 3.17 cm.) × 2. The deposition was performed in beakers, having a capacity of 400 c.c., a height of 10 cm. and 7.6 cm. in diameter. The deposit was bright and adherent and, although the amount of free acid present was very small, the metal was not spongy. The results obtained were in close accord with those assigned by theory, according to the law that the amount of metal deposited in a given time is proportional to the strength of solution.

## II. THE INFLUENCE OF TEMPERATURE UPON THE PRECIPITATION OF COPPER.

| Copper taken in grams. | Copper deposited. | Free acid. | c.c. $H_2O$. | Time in hours. | Temperature in degrees Cent. |
|---|---|---|---|---|---|
| (1) .0211 | .0019 | none | 150 c.c. | 1 | 20° |
| (2) " | .0046 | " | " | " | 40° |
| (3) " | .0080 | " | " | " | 60° |
| (4) " | .0119 | " | " | " | 80° |

The area of the electrodes was (3.5 cm. × 3.8 cm.) × 2. Distance between poles 2.8 cm. The current gave 1.25 c.c. HO gas per minute. (1) was slightly spongy and had a slight deposit of basic green salt at the top. (3) was somewhat dark and slightly spongy at the top but adherent. The amount of metal deposited increased with rise of temperature, as follows : .0027 grams (20°–40°), .0034 grams (40°–60°), .0039 grams (60°–80°). The ratio of increase also rose with the temperature, being greatest between 60° and 80°. In the above series the determinations were made in neutral solutions ; in the following, 15 c.c. of formic acid was added.

| Copper taken. | Copper deposited. | Free acid. | $H_2O$. | Time. | Temperature in degrees Cent. |
|---|---|---|---|---|---|
| Grams. | Grams. | c.c. | c.c. | Hours. | |
| (1) .1057 | .0104 | 15 | 150 | $\frac{1}{2}$ | 20° |
| (2) " | .0164 | " | " | " | 40° |
| (3) " | .0237 | " | " | " | 60° |
| (4) " | .0319 | " | " | " | 80° |

The distance between the poles was 2.9 cm., area of electrodes (3.5 cm. × 3.8 cm.) × 2, current strength 7.5 c.c. OH gas per minute. The ratio of increase was: (20°–40°) .0060 grams, (40°–60°) .0073 grams, (60°–80°) .0082 grams. A comparison between the two series of results would indicate that the presence of dilute free acid in moderate quantity exercises no material influence on the amount of metal deposited, even at elevated temperatures.

## III. The Influence of Pole Separation upon the Precipitation of Copper.

| | Copper taken. | Copper deposited. | $H_2O$. | Time. | Distance between electrodes. |
|---|---|---|---|---|---|
| | Grams. | Grams. | c.c. | Hours. | cm. |
| (1) | .1974 | .0133 | 700 | 1 | 1.58 |
| (2) | " | .0106 | " | " | 3.16 |
| (3) | " | .0093 | " | " | 4.75 |
| (4) | " | .0084 | " | " | 6.33 |
| (5) | " | .0078 | " | " | 7.91 |
| (6) | " | .0073 | " | " | 9.50 |
| (7) | " | .0064 | " | " | 12.66 |

The area of the electrodes was (3.8 cm. × 3.48 cm.) × 2, free acid present 10 c.c. (1) was slightly spongy. (7) was very close to the edge of the dish. The diminution was (1–2) .0027 grams, (2–3) .0013 grams, (3–4) .0009 grams, (4–5) .0006 grams, (5–6) .0005 grams. The current gave 1.75 c.c. OH gas per minute.

The foregoing experiments were performed in a crystallizing dish 15.2 cm. by 7.6 cm., with a capacity of 900 c.c. In the following series the determinations took place in a beaker 10 cm. in height by 7.6 cm. in diameter.

| | Copper taken. | Copper deposited. | $H_2O$. | Time. | Distance of electrodes. |
|---|---|---|---|---|---|
| | Grams. | Grams. | c.c. | Hours. | cm. |
| (1) | .1434 | .0302 | 200 | 1 | 1.58 |
| (2) | " | .0248 | " | " | 3.16 |
| (3) | " | .0208 | " | " | 4.75 |
| (4) | " | .0172 | " | " | 6.33 |

The current gave 1.75 c.c. oxyhydrogen gas per minute, area of electrodes (3.8 cm. × 3.16 cm.) × 2, free acid present 5 c.c. The diminution was (1–2) .0054 grams, (2–3) .0040 grams, (3–4) .0036 grams. In both series the rate of diminution rapidly lessened as the distance between the electrodes increased.

Experiments made under conditions similar to the above, except that no free acid was present, were failures, the deposits being exceedingly spongy.

## I. INFLUENCE OF DILUTION UPON THE PRECIPITATION OF CADMIUM.

| | Cadmium taken. | Cadmium deposited. | $H_2O$. | Free acid. | Time. | OH gas per minute. |
|---|---|---|---|---|---|---|
| | Grams. | Grams. | c.c. | c.c. | Hours. | c.c. |
| (1) | .0994 | .... | 100 | none | $\frac{1}{4}$ | 1.75 |
| (2) | .0497 | .... | " | " | $\frac{1}{2}$ | " |
| (3) | .0994 | .... | " | .5 | $\frac{1}{2}$ | " |
| (4) | " | .0218 | " | 5 | 1 | " |
| (5) | .0497 | .0109 | " | $\frac{5}{2}$ | " | " |
| (6) | .0248 | .0053 | " | $\frac{5}{4}$ | " | " |
| (7) | .0124 | .0026 | " | $\frac{5}{8}$ | " | " |
| (8) | .0062 | .0011 | " | $\frac{5}{16}$ | " | " |

In (1) and (2) no free acid was added, and in (3) only .5 c.c. was present; all three were failures. The amount of acid was then increased to 5 c.c., and the experiment repeated, all the other conditions remaining the same. The deposit was adherent and compact. The poles were 3.16 cm. apart, and had an area of (2.85 cm. × 3.8 cm.) × 2.

## II. INFLUENCE OF TEMPERATURE UPON THE PRECIPITATION OF CADMIUM.

| | Cadmium taken. | Cadmium found. | $H_2O$. | Free acid. | $H_2O$ gas per minute. | Temperature in degrees Cent. |
|---|---|---|---|---|---|---|
| | Grams. | Grams. | c.c. | c.c. | c.c. | |
| (1) | .1231 | .0240 | 150 | 10 | 1.75 | 20° |
| (2) | " | .... | " | " | " | 40° |
| (3) | .0497 | .0088 | 145 | 25 | " | 20° |
| (4) | " | .0110 | " | " | " | 40° |
| (5) | " | .0210 | " | " | " | 60° |
| (6) | " | .0257 | " | " | " | 80° |

(2) was very spongy and was covered with a white gelatinous deposit resembling cadmium hydrate. The amount of cadmium was then diminished more than half, while the amount of formic acid present was increased to 25 c.c. (5) and (6) were somewhat spongy but adherent. The increase was (20°-40°) .0022 grams, (40°-60°) .0100 grams, (60°-80) .0047 grams. The amount of metal deposited increased with rise of temperature, being greatest at 80°, but the greatest ratio of increase was at 60°, being almost five times greater than at 40°, and more than twice as great as at 80°. These results were so different from those obtained with copper that a second series of determinations was made, in which the amount of cadmium in the solution was reduced still more in order to insure a compact deposit at the higher temperatures. The results which were in close accord with those above are as follows:

| | Cadmium taken. | Cadmium deposited. | $H_2O$. | Free acid. | OH gas per minute. | Temperature in degrees Cent. |
|---|---|---|---|---|---|---|
| | Grams. | Grams. | c.c. | c.c. | c.c. | |
| (1) | .0198 | .0022 | 135 | 15 | 1.25 | 20° |
| (2) | " | .0031 | " | " | " | 40° |
| (3) | " | .0078 | " | " | " | 60° |
| (4) | " | .0102 | " | " | " | 80° |

### III. INFLUENCE OF POLE SEPARATION UPON THE PRECIPITATION OF CADMIUM.

| | Cadmium present. Grams. | Cadmium deposited. Grams. | $H_2O$. c.c. | OH gas per minute. c.c. | Separation of electrodes. cm. |
|---|---|---|---|---|---|
| (1) | .0994 | .... | 700 | 2.2 | 1.58 |
| (2) | " | .0059 | " | 1.25 | " |
| (3) | " | .0023 | " | .8 | " |
| (4) | " | .0009 | " | " | 3.16 |
| (5) | " | .0002 | " | " | 6.33 |
| (6) | " | .... | " | " | 12.66 |

Although 20 c.c. of free formic acid was present in (1) and (2) both were failures, the latter, though it was weighed, being merely approximate, some particles having been washed off. The current was then reduced to .8 c.c. HO gas per minute. Adherent deposits were obtained, but in such small quantity that (5) yielded only a trace. The ratio of diminution was (3–4) .0014 grams, (4–5) .0007 grams. Area of electrodes (3.8 cm. × 3.48 cm.) × 2, time 1 hour, free acid present 10 c.c.

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### I. INFLUENCE OF DILUTION UPON THE PRECIPITATION OF ZINC.

| | Zinc present. Grams. | Zinc deposited. Grams. | $H_2O$. c.c. | Free acid. c.c. | OH gas per minute. c.c. |
|---|---|---|---|---|---|
| (1) | .1250 | .0300 | 100 | .5 | 1.75 |
| (2) | .0625 | .0155 | " | .25 | " |
| (3) | .1250 | .0173 | " | 5. | " |
| (4) | .0625 | .0083 | " | $\frac{5}{2}$ | " |
| (5) | .0313 | .0043 | " | $\frac{5}{4}$ | " |
| (6) | .0156 | .0028 | " | $\frac{5}{8}$ | " |

(1), (2), (5) and (6) were spongy, especially the last. Distance between the poles 3.16 cm. Area (2.85 cm. × 3.8 cm.) × 2, time one hour.

### II. INFLUENCE OF TEMPERATURE UPON THE PRECIPITATION OF ZINC.

| | Zinc taken. Grams. | Zinc deposited. Grams. | $H_2O$. c.c. | Free acid. c.c. | OH gas per minute. c.c. | Temperature in degrees Cent. |
|---|---|---|---|---|---|---|
| (1) | .1539 | .... | 150 | 10 | .3 | 20° |
| (2) | .1026 | .0145 | " | " | 1.8 | " |
| (3) | .1026 | .... | " | " | " | 40° |
| (4) | .0205 | .... | " | none | .3 | 20° |
| (5) | " | .... | " | " | .8 | " |
| (6) | " | .... | " | " | 1.3 | " |
| (7) | .0513 | .0029 | 145 | 5 | .9 | " |
| (8) | " | .0019 | " | " | " | 40° |
| (9) | " | .0010 | " | " | " | 60° |
| (10) | " | .... | " | " | " | 80° |
| (11) | " | .... | " | " | " | " |
| (12) | " | .... | " | none | " | " |

(1), (3), (5) and (6) were very spongy. In (4), (10) and (11) no deposition of metal took place, (12) was spongy and was covered with a white coating of zinc hydrate. At 80° no metal was deposited in the presence of free acid provided the current was not too strong. The ratio of decrease with rise of temperature was (20°–40°) 10 grams, (40°–60°) 9 grams, (60°–80°) no deposit. The distance between the poles was 2.85 cm. Area of electrodes (3.8 cm. × 3.16 cm.) × 2. Duration of experiment, one hour.

### III. INFLUENCE OF POLE SEPARATION UPON THE PRECIPITATION OF ZINC.

| | Zinc taken. | Zinc deposited. | Free acid. | $H_2O$. | OH gas per minute. | Time. | Distance between poles. |
|---|---|---|---|---|---|---|---|
| | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. | cm. |
| (1) | .0102 | .0046 | 10 | 700 | 1.9 | 3 | 1.58 |
| (2) | " | .0023 | " | " | " | " | 3.16 |
| (3) | " | .0015 | " | " | " | " | 6.33 |
| (4) | " | .0003 | " | " | " | " | 12.66 |

Area of electrodes (3.8 cm. × 3.48 cm.) × 2. The deposit was firm and compact. Compared with the results obtained with copper and cadmium, the result in (4) is too low.

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### LEAD (DETERMINED ELECTROLYTICALLY).

On account of tendency of lead and manganese to separate in the form of peroxide at the positive pole, it was deemed advisable to make a series of experiments on the metals themselves before attempting to effect their separation. The results were as follows :

| | Lead taken. | Lead found. | Free acid. | $H_2O$. | OH gas per minute. | Time. | Difference from theory. |
|---|---|---|---|---|---|---|---|
| | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. | |
| (1) | .1153 | .... | 5 | 100 | 1. | 16 | .... |
| (2) | " | .... | 20 | " | 1.2 | 48 | .... |
| (3) | " | .... | 5 | " | 2.8 | 3 | .... |
| (4) | " | .... | 20 | " | " | 16 | .... |

50 c.c. of a lead formate solution were used in each of the above experiments. In all of them, the lead was deposited in a spongy state at the kathode with more or less peroxide on the positive pole.

As the moist metal deposited on the kathode rapidly oxidizes, even when adherent and compact, the results obtained are invariably too high and in practice it is customary to estimate lead as peroxide on the anode securing its deposition in that form by the addition of nitric acid to the solution. The results obtained with free formic acid, as given above, were not such as to justify attempting its separation from either copper, cadmium or zinc.

### Manganese (Determined Electrolytically).

| | Manganese taken. | Manganese found. | Free acid. | $H_2O$. | OH gas per min. | Time. | Difference from theory. |
|---|---|---|---|---|---|---|---|
| | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. | Percentage. |
| (1) | .0554 | .0552 | 5 | 100 | 2.2 | 17 | —.36 |
| (2) | " | .0556 | " | " | 2.7 | 18 | +.36 |
| (3) | .1108 | .1101 | 20 | " | 2.8 | 24 | —.63 |
| (4) | .0554 | .... | 30 | " | 1.6 | 16 | .... |

The platinum dish was made the anode, the wire spiral serving as the negative pole. With small quantities of free acid, (1) and (2), considerable peroxide of manganese, separated on both poles, with larger quantities, (3) (4), only very slight traces were found on the kathode. The deposition in (4) was not complete. The peroxide formed a black, lustrous coating on the dish, adherent while moist, but scaling off upon being heated.

The manganese which separated on the kathode was removed by means of a small piece of filter paper, which was ignited and the ash added to the contents of the dish, which was then raised to an intense heat in order to convert the peroxide of manganese into $Mn_3O_4$, in which form it was finally weighed. Traces of Mn were found in solution (3).

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## ELECTROLYTIC SEPARATIONS.

### Cadmium from Manganese.

| | Cadmium taken. | Manganese taken. | Cadmium found. | Free acid. | $H_2O$. | OH gas per min. | Time. | Difference from theory. |
|---|---|---|---|---|---|---|---|---|
| | Grams. | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. | Percentage. |
| (1) | .0497 | .0554 | .0425 | 35 | 75 | .8 | 16 | .... |
| (2) | " | " | .0440 | " | " | 1 | " | .... |
| (3) | " | " | .0498 | 25 | " | 2.7 | 17 | +.20 |
| (4) | .0511 | " | .0509 | 20 | " | 2.4 | 19 | —.39 |
| (5) | " | " | .0697 | 5 | 150 | 2.7 | 18 | .... |
| (6) | .1022 | .1108 | .1098 | 20 | 75 | 2.5 | 17 | .... |
| (7) | " | " | .... | 40 | 150 | 2.8 | 45 | ... |
| (8) | .0511 | .0554 | .0514 | 30 | 75 | 2.7 | 18 | +.58 |

In the preliminary experiments on manganese alone, it was found that the presence of 20 c.c. of free acid was sufficient to prevent the deposition of any peroxide on the kathode, except in the very slightest traces; but the presence of cadmium in the solution seemingly had a contrary effect, as the presence of even 40 c.c. of acid failed to prevent the separation of traces of manganese on the negative pole (7). In (5), to which 5 c.c. of free acid had been added, the deposit of peroxide of manganese upon the negative pole was almost five times greater than in a solution of manganese to which no cadmium had been added, all the other conditions being the same. In all the above experiments the platinum dish was used as the anode, the platinum crucible serving as the negative electrode. In (1) and (2) the cadmium was not completely deposited. Traces of cadmium were found in (6) and (7). More or less manganese was found in all the deposits, but only in traces in the presence of more than 20 c.c. of free acid ; (4) and (7) were very spongy ; the others were slightly so at the periphery of the crucible, but adherent. The best results were obtained by fulfilling the conditions described in (3), (4) and (8) ; but to obtain a compact deposit of cadmium free from all traces of manganese, it is evident that the amount of free acid must be increased and the poles separated. Under these conditions, a stronger current must be employed than that furnished by the battery of "crowfoot" cells, with which my experiments were carried on.

### ZINC FROM MANGANESE.

| Zinc taken. | Manganese taken. | Zinc found. | Free acid. | $H_2O$. | OH gas per min. | Time. | Difference from theory. |
|---|---|---|---|---|---|---|---|
| Grams. | Grams. | | c.c. | c.c. | c.c. | Hours. | Percentage. |
| (1) .0562 | .0554 | .... | 30 | 90 | 2.9 | 17 | .... |
| (2) " | " | .... | 20 | 100 | " | 16 | .... |

The dish served as anode, the crucible as kathode. Both were failures ; the zinc being spongy and containing $MnO_2$ and not entirely precipitated.

### COPPER FROM ZINC.

| Copper taken. | Zinc taken. | Copper found. | Free acid. | $H_2O$. | OH gas per min. | Time. | Difference from theory. |
|---|---|---|---|---|---|---|---|
| Grams. | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. | Percentage. |
| (1) .1074 | .0818 | .... | 5 | 100 | 1.8 | 46 | .... |
| (2) " | " | .... | " | " | 1.2 | 17 | .... |
| (3) " | .1124 | .... | " | " | .8 | 16 | .... |
| (4) " | .0818 | .1072 | 15 | " | " | " | —.18 |
| (5) " | " | .1073 | 20 | " | " | 17 | —.09 |
| (6) .0987 | " | .0990 | " | " | " | " | +.30 |
| (7) " | " | .0984 | " | " | .6 | 16 | —.30 |
| (8) .1057 | .1006 | .1052 | 15 | " | .8 | " | —.47 |
| (9) " | " | .1061 | " | " | " | 18 | +.37 |
| (10) " | " | .1058 | " | 150 | " | " | +.09 |
| (11) " | " | .1059 | 20 | " | " | 19 | +.18 |
| (12) " | " | .1053 | " | " | " | 21 | —.37 |
| (13) " | " | .1060 | " | " | " | 16 | +.28 |

As will be seen from the above, it was possible to separate copper free from zinc, except the slightest traces, by using a weak current in solutions to which 15–20 c.c. of free formic acid had been added. By employing stronger currents, or diminishing the amount of free acid, the copper was deposited admixed with considerable quantities of zinc. If the necessary precautions are observed no zinc will be deposited, and the copper will be compact and adherent.

### CADMIUM FROM ZINC.

| | Cadmium taken. | Zinc taken. | Cadmium found. | Free acid. | $H_2O$. | OH gas per min. | Time. | Difference from theory. |
|---|---|---|---|---|---|---|---|---|
| | Grams. | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. | Percentage. |
| (1) | .1231 | .0818 | .... | 15 | 100 | .2 | 17 | .... |
| (2) | " | " | .1229 | 30 | " | .8 | 19 | —.16 |
| (3) | " | " | .1234 | " | " | " | 43 | +.24 |
| (4) | " | " | .1426 | 35 | " | 1.25 | 16 | +15.83 |
| (5) | " | " | .1233 | 30 | " | .8 | " | +.16 |
| (6) | .0492 | .0409 | .... | 15 | " | " | " | .... |
| (7) | " | " | .0842 | " | " | 1.25 | " | .... |
| (8) | .0984 | .1026 | .0322 | 35 | " | .8 | 19 | .... |
| (9) | .0492 | .0409 | .0494 | 15 | " | " | 41 | +.40 |
| (10) | .0984 | .1026 | .1735 | 35 | 75 | 1.25 | 17 | .... |
| (11) | " | " | .0722 | " | " | .8 | " | .... |
| (12) | " | " | .0982 | 25 | 125 | " | 18 | —.20 |
| (13) | " | " | .0985 | " | " | " | 16 | +.10 |
| (14) | .1004 | .1006 | .1001 | " | " | 1. | 48 | —.29 |
| (15) | " | " | .0993 | " | " | " | 17 | —1.09 |
| (16) | " | " | .1000 | " | " | " | " | —.39 |
| (17) | " | " | .1001 | " | " | " | 18 | —.29 |
| (18) | " | " | .1008 | " | " | " | 20 | +.39 |
| (19) | " | " | .0999 | " | " | " | 16 | —.49 |

The first seven determinations were made in a platinum dish weighing about 67 grams; the remainder in a much larger dish weighing 117 grams. The results obtained with the latter were not as satisfactory as with the smaller dish (2) (3) (5), although a qualitative examination of the deposit and solution proved that the separation was complete (9) (12–19). The distance between the poles materially influences the results. In (6) the positive pole was in close contact with the dish; the cadmium contained zinc. In (9) the conditions were similar in all respects to the preceding, except that the distance between the poles was 2.5 cm.; the deposit was free from zinc. With .2 c.c. OH gas per minute, only a small quantity of the cadmium was separated (1), the greater portion remaining in solution. With 1.25 c.c., on the other hand, the zinc was deposited as a dark-gray coating upon the cadmium (7), even in the presence of 35 c.c. of free acid (10). In solutions containing .10 grams of each metal a current of .8–1 c.c. HO gas per minute sufficed to secure a satisfactory deposit in the presence of 25 c.c. formic acid (12–19). With smaller quantities of metal (9) 15 c.c. of free acid was sufficient. The deposits in the above experiments were adherent and compact. There was no tendency to sponginess even in deposits containing large quantities of zinc.

### Copper from Cadmium.

| Copper taken. | Cadmium taken. | Metal deposited. | Free acid. | $H_2O$. | OH gas per min. | Time. | Difference from theory. |
|---|---|---|---|---|---|---|---|
| Grams. | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. | Percentage. |
| .1074 | .0984 | .2061 | 10 | 100 | .8 | 17 | .... |

This result was not unexpected, considering what we have already learned in regard to the behavior of these metals. The deposit was very dark and spongy. Both metals were completely precipitated. Two subsequent experiments were equally unsatisfactory; in one the current was reduced to .3 c.c. OH gas per minute; in the other, 25 c.c. of formic acid was added. No separation was effected.

### Copper from Iron.

| | Copper taken. | taken. | Copper found. | Free acid. | $H_2O$. | OH gas per min. | Time. | Difference from theory. |
|---|---|---|---|---|---|---|---|---|
| | Grams. | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. | Percentage. |
| (1) | .1057 | .1248 | .1035 | 25 | 125 | .8 | 20 | .... |
| (2) | " | " | .... | " | 150 | " | 42 | .... |
| (3) | " | " | .1019 | " | " | " | 19 | .... |
| (4) | " | " | .0999 | " | " | " | 17 | .... |
| (5) | " | " | .1014 | " | " | " | 18 | .... |

Although free acid was present in considerable quantity (25 c.c.), the formate of iron in the solution was decomposed with the formation of ferric hydrate, which separated as a light yellow froth on the surface of the solution. It also formed crusts at the edge of the copper deposit, which adhered to the dish with such tenacity that all attempts at removal by mechanical means were failures. Mere washing was without avail, and more energetic measures resulted in a loss of copper, (1) and (4). Dilute hydrochloric acid was added to (2); but, while it dissolved traces of copper, it failed to remove the deposit of iron hydrate. Dilute sulphuric acid was also tried without success (3). In the final determination 20 c.c. of concentrated oxalic acid was added at the end of seventeen hours and the current allowed to act one hour longer. The froth floating on the surface was dissolved, but the deposit on the copper was not appreciably affected. Except at the periphery the copper had a bright metallic lustre and was firm and adherent.

### Cadmium from Iron.

| | Cadmium taken. | Iron taken. | Cadmium found. | Free acid. | $H_2O$. | OH gas per min. | Time. |
|---|---|---|---|---|---|---|---|
| | Grams. | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. |
| (1) | .0984 | .1248 | .0996 | 25 | 150 | .8 | 23 |
| (2) | " | " | .1021 | " | " | " | 45 |

The same trouble was experienced here. Similar attempts were made to remove the iron, but without success. In the first of the above determinations 10 c.c. of oxalic acid solution was added before the conclusion of the experiment; in the latter, 20 c c. of the same solution.

The hydrate of iron in the solution disappeared, but adherent crusts still remained on the surface of the cadmium.

### ZINC FROM IRON.

Several tentative experiments were made, but as the iron showed the same tendency to separate on the sides of the dish, as in the preceding determinations, they were not continued.

### COPPER FROM COBALT.

| | Copper taken. Grams. | Cobalt taken. Grams. | Copper found. Grams. | Free acid. c.c. | $H_2O$. c.c. | OH gas per min. c.c. | Time. Hours. | Difference from theory. Percentage. |
|---|---|---|---|---|---|---|---|---|
| (1) | .1101 | .1080 | .1105 | 100 | 175 | 1 | 21 | +.36 |
| (2) | “ | “ | .1095 | “ | “ | “ | 17 | —.54 |
| (3) | “ | “ | .1097 | “ | “ | “ | “ | —.36 |
| (4) | “ | “ | .1107 | “ | “ | “ | 18 | +.54 |
| (5) | “ | “ | .1098 | “ | “ | 1.2 | 17 | —.27 |
| (6) | “ | “ | .1097 | “ | “ | 1 | 16 | —.36 |

On attempting to prepare a solution of cobaltous formate for the above determinations, it was found that the salt made according to the method already described was not readily soluble in water. The solution was therefore prepared by double decomposition as follows: 500 c.c. of water containing 6.563 grams of sodium formate was mixed with an equal amount of water in which 8.728 grains of cobalt chloride had been dissolved.

Of this solution 50 c.c. was taken, containing .1080 grams of cobalt. The distance between the poles was 3.8 cm. except (1) and (4) in which it was 2.8 cm. Both of the latter were spongy; the others slightly so. As the conditions, otherwise, were similar, the difference in the character of the deposits was apparently due to the separation of the poles. Traces of cobalt were found in all the copper deposits. The copper was all out except in (3), (5) and (6), in which the solutions were colored yellowish brown on the addition of hydrogen sulphide.

The copper deposit was dark in color and adherent, although not very compact on the bottom of the dish.

### COPPER FROM NICKEL.

| | Copper taken. Grams. | Nickel taken. Grams. | Copper found. Grams. | Free acid. c.c. | $H_2O$. c.c. | OH gas per min. c.c. | Time. Hours. | Difference from theory. Percentage. |
|---|---|---|---|---|---|---|---|---|
| (1) | .1101 | .1028 | .1095 | 75 | 175 | 1 | 20 | —.54 |
| (2) | “ | “ | .1097 | 100 | “ | “ | 17 | —.36 |
| (3) | “ | “ | “ | “ | “ | “ | 18 | —.36 |
| (4) | “ | “ | .1098 | “ | “ | 1.2 | 17 | —.27 |
| (5) | “ | “ | .1096 | “ | “ | 1 | “ | —.46 |
| (6) | “ | “ | .1098 | “ | “ | “ | “ | —.27 |

The same trouble was experienced in preparing a satisfactory solution of pure nickel formate as with cobalt and it was found advisable to prepare the solution by double decomposition in the same way as the latter salt, 500 c.c. of this solution contained 8.3077 grams of nickel chloride and 6.2469 grams of sodium formate. In both cases a slight excess of sodium formate was used. The copper contained traces of nickel and slightly colored the solution when tested with hydrogen sulphide. The conditions were similar to those given under cobalt and the results were quite as satisfactory. The copper was bright and compact.

### Cadmium from Cobalt.

| | Cadmium. taken. Grams. | Cobalt. taken. Grams. | Cadmium found. Grams. | Free acid. c.c. | $H_2O$. c.c. | OH gas per min. c.c. | Time. Hours. | Difference from theory. Percentage. |
|---|---|---|---|---|---|---|---|---|
| (1) | .0984 | .1080 | .... | 25 | 100 | .5 | 22 | .... |
| (2) | “ | “ | .... | “ | 150 | .8 | 45 | .... |
| (3) | “ | “ | .... | 50 | “ | 1.5 | 18 | .... |

It was naturally expected that cadmium would be completely precipitated from cobalt and nickel by employing a weak current, but from an examination of the above results, it will be seen that a separation was not accomplished.

Even with a current of 1.5 c.c. OH gas per minute, the cadmium failed to deposit completely and was contaminated with cobalt (3). (1) was very spongy and the solution still contained cadmium at the expiration of 22 hours. The current was then increased and allowed to act for 45 hours (2). Cadmium was found in the solution, cobalt in the deposit. The distance between the electrodes was 2.8 cm.

### Cadmium from Nickel.

| | Cadmium taken. Grams. | Nickel taken. Grams. | Cadmium found. Grams. | Free acid. c.c. | $H_2O$. c c. | OH gas per min. c.c. | Time. Hours. | Difference from theory. Percentage. |
|---|---|---|---|---|---|---|---|---|
| (1) | 0984 | .1028 | .0758 | 35 | 150 | .5 | 19 | .... |
| (2) | “ | “ | .1045 | “ | “ | 1.5 | 21 | .... |
| (3) | “ | “ | .1348 | 50 | 125 | 1.5 | 17 | .... |

The results were quite as unsatisfactory as with cobalt. Cadmium was found in all three solutions, and more or less nickel was found in the deposits. In (3) the nickel came out as a gray deposit on the cadmium. The deposit was firm and adherent, although dark in color. The distance between the electrodes was 2.5 cm., except (3), in which the pole separation was 2.8 cm.

### Zinc from Cobalt.

| | Zinc present. Grams. | Cobalt present. Grams. | Zinc found. Grams. | Free acid. c.c. | $H_2O$. c.c. | OH gas per min. c.c. | Time. Hours. | Difference from theory. Percentage. |
|---|---|---|---|---|---|---|---|---|
| (1) | .1006 | .1080 | .... | 50 | 175 | 3 | 17 | .... |
| (2) | “ | “ | .... | 100 | “ | 5 | 18 | .... |

### ZINC FROM NICKEL.

| | Zinc present. | Nickel present. | Zinc found. | Free acid. | $H_2O$. | OH gas per min. | Time. | Difference from theory. |
|---|---|---|---|---|---|---|---|---|
| | Grams. | Grams. | Grams. | c.c. | c.c. | c.c. | Hours. | Percentage. |
| (3) | .1006 | .1028 | .... | 50 | 175 | 2.7 | 18 | .... |
| (4) | " | " | .... | 100 | " | 5 | " | .... |

(2) and (4) were performed under similar conditions. The distance between the poles was 2.2 cm. The current was generated by a battery of Bunsen cells. Even with a current of 5 c.c. gas per minute zinc was found in the solution in traces, while considerable quantities of cobalt and nickel separated as a coating upon the cadmium. (1) and (3) were also failures. A separation was not obtained even approximately.

### SUMMARY.

As a result of the foregoing experiments, it was found that the amount of copper, cadmium or zinc deposited in a given time was proportional to the strength of the solution, and that the presence of free acid in moderate quantity did not materially affect the result.

Increasing the distance between the poles resulted in diminishing the amount of metal deposited, but the rate of decrease diminished as the distance between the electrodes increased.

Elevation of temperature caused an increase in the amount of metal deposited, the rate of increase being greatest at 80° in neutral and acid copper solutions, and at 60° in cadmium solutions containing free acid. On the other hand, the amount of zinc deposited in solutions, to which free acid had been added, diminished as the temperature rose, nothing being deposited at 80°.

Attempts to secure compact and adherent deposits of cadmium and zinc in neutral solutions were failures.

In acid solution copper and cadmium separated completely and satisfactorily. The zinc deposits were spongy, but the precipitation was complete.

Lead was mainly deposited on the negative pole, both in neutral and acid solutions. Manganese was precipitated on both poles, but the amount of peroxide separating on the kathode was reduced to mere traces by the presence of free acid.

The following separations were satisfactorily accomplished : copper from zinc, cobalt and nickel and cadmium from zinc and manganese.

Attempts to deposit copper in the presence of iron and cadmium, and zinc in the presence of iron, cobalt and nickel, were successful. Nor was it possible in the presence of the last three metals named to estimate cadmium.

In conclusion, I wish to express my obligations to Prof. Edgar F. Smith, at whose suggestion the work was undertaken. To his supervision and advice is largely due whatever value may attach to these results.